

FY 1996 End of Fiscal Year Letter  
(01 Oct 1995 - 30 Sep 1996)

ONR CONTRACT INFORMATION

Contract Title: Environmental Integrity of the Coating Metal Interface - Novel Non-VOC Technology

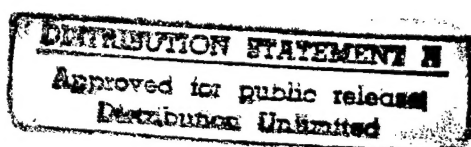
Performing Organization: Rockwell International Science Center

Principal Investigator: Martin W. Kendig

Contract Number: N00014-92-C-0215

R and T Project Number:

ONR Scientific Officer: A. John Sedriks



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#### A. Scientific Research Goals

Environmental concerns demand a reduction if not total elimination of the emissions of volatile organic compounds (VOCs) from all manufacturing processes. This study seeks to identify the critical factors which will lead to the optimization of a non-VOC epoxy coating and ultimately to produce a prototype non-VOC marine primer. Our goal is to develop an ambient temperature curable epoxy coating that is fully compatible with supercritical CO<sub>2</sub> (SCF CO<sub>2</sub>) without VOC and can meet the performance requirements at least equivalent to Mil Spec 24441. Our efforts have been focused on (i) studying the relationship between the structure of amine curing agent and its reactivity with SCF CO<sub>2</sub> and (ii) identifying an amine curing agent that can reversibly release CO<sub>2</sub> at a rate fast enough to allow the formation of a coating that is uniform and void free. Our approach entails the deposition of the epoxy resin from a supercritical CO<sub>2</sub> (SCF CO<sub>2</sub>) solution.

#### B. Significant Results in FY 1996

A significant result that we achieved in FY 1996 has been completion of experiment and tests designed to directly compare our best formulation of an epoxy coating sprayed using supercritical carbon dioxide (SCF CO<sub>2</sub>) with a MIL P 24441 chromate-containing primer. The supercritical coating contained no chromates, but rather contained a phosphate/molybdate corrosion inhibiting pigment. In addition, we have been actively investigating the development of a room temperature-cured supercritical CO<sub>2</sub> deposited epoxy.

##### Comparison of SCF CO<sub>2</sub> Epoxy with MIL P 24441

Figures 1 and 2 show the time dependence for coating and corrosion resistance and corrosion potential for the non-chromate SCF CO<sub>2</sub> applied coating and two MIL P24441 coatings on sandblasted steel substrates. The MIL 24441 coating thicknesses are comparable or greater than that for the SCF CO<sub>2</sub> epoxy of nearly comparable thickness. The SCF CO<sub>2</sub> coating exhibits much higher corrosion resistance for the first 500 h than either of the MIL P 24441 coated steels. We can conclude that the SCF CO<sub>2</sub> epoxy performs at least as well as the comparable MIL P 24441. The comparable to superior performance of the SCF CO<sub>2</sub> also appears from ASTM B117 salt fog test results as shown in Figure 3. Scribe creep back and onset of corrosion for coatings of comparable thickness appear to be the same. Note that the SCF CO<sub>2</sub> coating used in these comparisons was a 80 C cured coating. The SCF CO<sub>2</sub> epoxy also showed superior resistance to cathodic disbonding as compared to the MIL P 24441. Effort continues to make a room temperature cured epoxy.

##### Room Temperature Cure - Progress

(i) In collaboration with Phasex Corporation, we determined the cure rates of epoxy-amine mixtures in SCFCO<sub>2</sub> and found that the cure rate was considerably slowed in the presence of CO<sub>2</sub> as compared with that in the absence of CO<sub>2</sub>, indicating that SCFCO<sub>2</sub> is remarkably effective in preventing the advancement of epoxy-amine reaction by forming the corresponding amine- CO<sub>2</sub> adducts (presumably carbamates).

(ii) We prepared various secondary amines by treating primary amines with epoxies and acrylonitrile and determined their reactivity toward epoxies and CO<sub>2</sub> by measuring the viscosity as well as by monitoring with FTIR. Several amines demonstrated the fast release of CO<sub>2</sub> upon exposure to air. However, those amines also reacted slowly with epoxies and required a longer time to complete the cure. Attempts are being made to identify a catalyst to accelerate the cure.

#### C. Plans for FY 1997

We have requested a no cost extension on this program in order to finish final reporting in the form of a series of papers now in preparation and to perform several coating tests designed to improve the adhesion of the room temperature cured SCF CO<sub>2</sub> epoxy coatings.

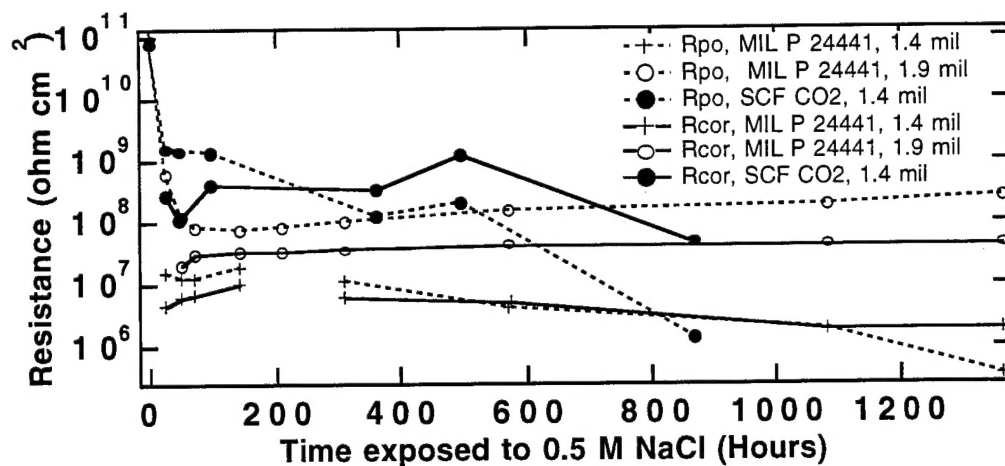


Figure 1. Coating resistance (Rpo) and corrosion resistance (Rcor) for samples exposed to 0.5 M NaCl (air equilibrated).

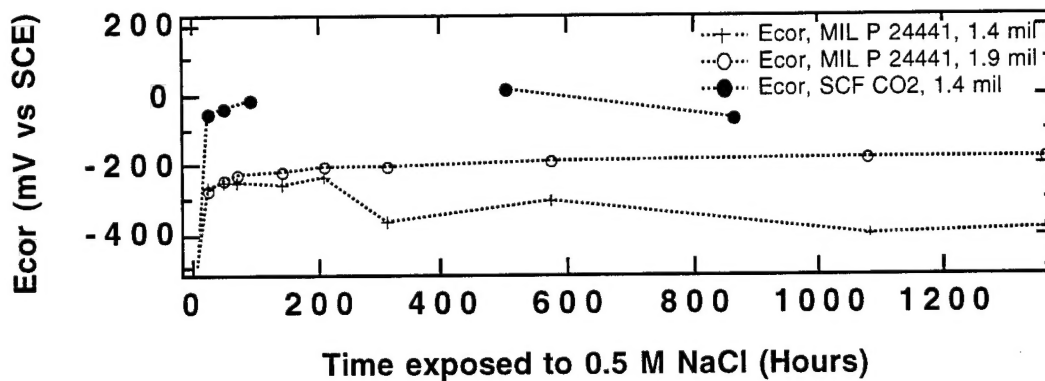


Figure 2. Corrosion potential for samples of Figure 1.

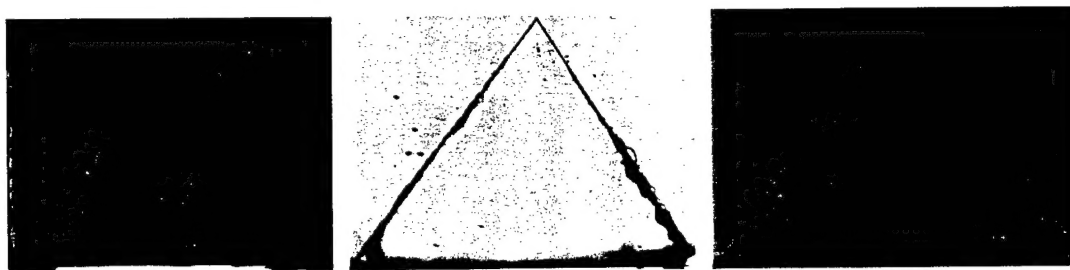


Figure 3. Photographs of the scribed portion of test coupons (sand-blasted carbon steel) exposed to 1000 h of ASTM B117 salt fog: (a) MIL P 24441 (1.3 mil), (b.) SC F CO<sub>2</sub> epoxy (1.4 mil), (c.) MIL P 24441 (1.9 mil).

#### D. List of Publications/Reports/Presentations

##### 1. Papers Published in Refereed Journals

none

##### 2. Non-Refereed Publications and Published Technical Reports

##### 3. Presentations

###### a. Invited

M. Kendig, S. Jeanjaquet and R. Brown, "Rapid Electrochemical Assessment of Automotive Paint", Corrosion '96 Research in Progress Symposium, March 1996.

M. Kendig, "Overview of Non-Electrochemical Techniques for Coating Assessment", 2nd Workshop on Quantitative Methods for Predicting Coating Performance, Naval Surface Warfare Center, Carderock Division, Annapolis, MD, November 1995

M. Kendig, "Corrosion Inhibition of Cu-Rich Al Alloys", Materials Science Seminar, The Pennsylvania State University, University Park, PA, July 1996.

###### b. Contributed

M. Kendig, M. Cunningham, S. Jeanjaquet and H. Jensen, "Non-Chromate Corrosion-Inhibiting Pigments", Symposium on Environmentally Acceptable Inhibitors and Coatings, 188th Meeting of the Electrochemical Society, October 1995.

M. Kendig, S. Jeanjaquet, D. Hardwick and M. Cunningham, "Corrosion Inhibition of theta Al<sub>2</sub>Cu by Borates", Electrochemical Society, San Antonio, October 1996.

M. Kendig, "Life Prediction of Automotive Coatings", 2nd Workshop on Quantitative Methods for Predicting Coating Performance, Naval Surface Warfare Center, Carderock Division, Annapolis, MD, November 1995

##### 4. Books (and sections thereof)

#### E. List of Honors and Awards

none

# H. Summary of FY 1995 Publications/Patents/Presentations/Honors/Participants

	ONR	Non-
ONR		
a. Number of Papers Submitted to Refereed Journals but not yet published	1	3
b. Number of Papers Published in Refereed Journals	0	0
c. Number of Books or Chapters Submitted but not yet published	0	0
d. Number of Books or Chapters Published	0	0
e. Number of Printed Technical Reports and Non-Refereed Papers	0	0
f. Number of Patents filed	0	0
g. Number of Patents granted	0	0
h. Number of Invited Presentations at Workshops of Professional Societies	1	2
i. Number of Contributed Presentations at Workshops or Professional Society Meetings	0	3
j. Honors/Awards/Prizes for Contract/Grant Employees	0	0
k. Number of Graduate Students and Post-Docs	0	0
l. Number of Female or Minority PIs or Co-PIs	0	0